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(56) Documents Cited
EP 0501257 A1 **US 4132892 A**
Nuclear Instruments and Methods in Physics Research A Vol.415, No.3, October 1998, pages 653-656
Molecular Materials Vol.11, No.1-2, 1998, pages 131-134
Chinese Physics Letters Vol.10, No.8, 1993, pages 453-455
Zeitschrift für Physik D Vol.29, No.1, 1994, pages 53-59
Hyperfine Interactions Vol.99, No.1-3, February 1996, pages 175-191

(58) Field of Search
UK CL (Edition V) **H1D**
INT CL^7 **H01J**
Other: **Online: WPI, EPODOC, JAPIO, INSPEC**

(54) Abstract Title
Fullerene ion gun

(57) A fullerene ion gun capable of producing a beam of C_{60} ions, which is pulsed, mass filtered and has sufficient intensity for use as a probe in static time-of-flight secondary ion mass spectrometry (S-TOF SIMS). C_{60} powder is held in a cylindrical reservoir 2, which is heated by a heater 4 in order to vaporize the powder. C_{60} vapour issues from a nozzle 5 into an ionization chamber enclosed by a grid 6. A circular filament 7 releases electrons which are accelerated by the grid 6 into the centre of the chamber, where fullerene ions are formed by electron bombardment of the C_{60} vapor. Ions are extracted by an electrode 8 and formed into a probe by electrostatic lens 9 and 12, which may be scanned over a sample by scanning electrodes 13. The gun also has components for mass filtering 14, pulsing the beam on and off 10, and includes a bend 11 for rejection of neutrals from the ion beam.

Figure 1

Schematic Cross Section of Fullerene Ion Gun

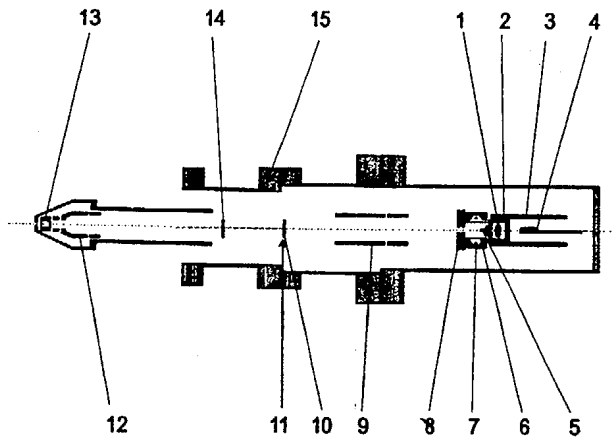

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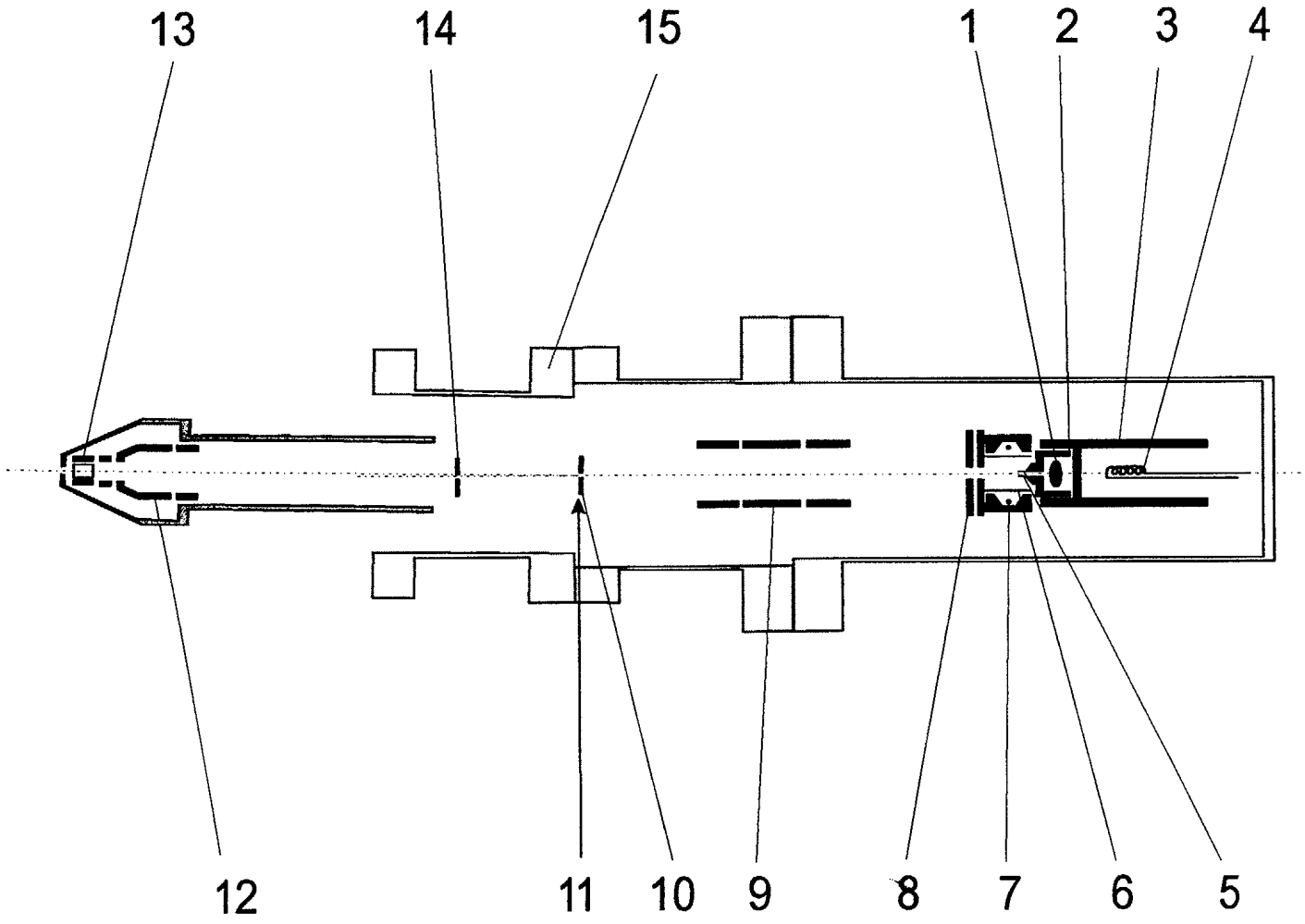
Figure 1

Schematic Cross Section of Fullerene Ion Gun



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FULLERENE ION GUN

This invention relates to an ion source and ion-optical column which will generate a focused beam of C_{60} ions and ions of other fullerene type molecules. Such an ion beam is suitable for a range of surface analytical techniques.

Ion guns are widely used in analysis and microfabrication techniques. They take a range of forms depending on the ion species which is required and the required spot shape of the ion beam. In an ion gun, ions are created by one of a range of ionising processes in an ion source and there are several different types of source that are conventionally used. Whatever the source, it is usually placed in close proximity to an extraction electrode to create a spray of ions from the source. These ions proceed along an optical column which contains electrostatic lenses, deflectors and other ion-optical devices to bring the ion beam to a focus at a particular point. Usually, this point lies on the surface of a sample to be analysed or on the surface of a work piece to be etched or to undergo a deposition process. The whole is contained inside a vacuum system. The extraction electrode and lenses are usually cylindrically symmetrical.

Such ion beams are used, for instance, in secondary ion mass spectrometry (SIMS) in which the focused ion beam is directed at a sample and causes secondary ions to be ejected from the sample. These secondary ions are collected into a mass analyser and a mass spectrum of a small area of the surface is produced. By scanning the primary ion beam over an area of the sample, typically using deflector plates to steer the beam in a line-and-flyback raster, an image of the distribution of a particular ion species can

be generated. With some types of ion source, the ion beam has a very small spot diameter and the spatial resolution of the ion image can be less then 0.1 micron. However, for many SIMS applications, a spot diameter of 1 micron is suitable.

When SIMS techniques are applied in organic chemistry, it is highly desirable that large intact molecules (or at least large fragments) are released from the sample surface under the impact of the primary ion species, to facilitate identification of the molecules in the mass spectrum. Atomic or light molecular ion beams conventionally used in the technique cause a very high degree of fragmentation of surface molecules. A C_{60} ion beam, the ions having mass 720 A.M.U., has the potential to reduce this fragmentation through its softer impact and thereby to enhance yields of large molecules from the surface.

A fullerene ion source suitable for SIMS applications must have sufficient intensity that the beam impinging on the sample can have a spot size in the range of 1 to 10 microns, whilst having sufficient ion current to produce an adequate yield of secondary ions from the surface.

One type of ion source is the electron bombardment source. Such a source is used for generation of ion beams from the atoms or molecules of a gas, usually a noble gas or oxygen. The gas is leaked into a vacuum chamber at low pressure. Some of the gas is lost directly to the vacuum system, but some enters an ionisation chamber where it is bombarded by low energy electrons to produce ions of the gas. Because the gas is leaked into the vacuum chamber from outside, the point of entry is often quite remote from the ionisation chamber for reasons of electrical isolation. The density of the

electron flux inside the ionisation chamber is limited by the rate of escape of electrons and their capture by surfaces at positive voltage.

According to the present invention, an electron bombardment ion source can be so constructed such that the gas is supplied through a nozzle directly into the centre of the ionisation chamber. The gas is fullerene vapour produced by the heating of a C_{60} powder reservoir situated near to the ionisation chamber. Furthermore, repellor electrodes are used to force electrons into orbits which repeatedly pass through the centre of the ionisation chamber. By these means, the source brightness becomes sufficient to allow the production of an ion beam with a sufficiently small spot size and sufficiently high current to be useful in SIMS applications.

In the present invention, ions are extracted from the source by an extraction electrode and are transported through the optical column to form a beam which is suited to imaging Time of Flight SIMS. For this application, the beam must be pulsed, mass filtered and scanned, and it must be free from neutral atoms.

A specific embodiment of the invention is shown in figure 1. The C_{60} powder (1) is contained in a cylindrical reservoir (2) which is itself held in a copper surround (3). This assembly is heated by a heater (4) to vaporise the C_{60} powder. C_{60} vapour issues from the nozzle (5) into the centre of the ionisation chamber which is enclosed by the grid (6). Outside the grid is a circular filament (7) This releases electrons which are then accelerated by the voltage on the grid into the centre of the chamber. The reservoir and grid are at high voltage, typically 20kV, in order that an ion beam can be produced with sufficient energy to have a small focus on a grounded sample. As

fullerene ions are formed by interaction with the electrons, an extraction electrode (8), at a potential of several hundred volts relative to the grid, attracts the ions for transportation into the optical column. The ions initially pass through an electrostatic lens (9) which forms a field image of the source in between a pair of deflector plates (10) which can be used to pulse the beam on and off. In order to reject neutrals from the beam, it is necessary that the beam path should bend, and in this embodiment, a 1° bend (11) is built into the column at the position of the deflector plates. The beam continues along the column and is focused onto the sample by a second lens (12) and is scanned over the sample by a set of scanning electrodes (13). The optical column includes a means of mass filtering the beam (14). The whole assembly is contained in a vacuum housing (15). Electrical connections to the various components enter the vacuum housing via vacuum feedthroughs (these connections not shown).

CLAIMS

1) A fullerene ion gun in which:
   a) fullerene vapour is introduced directly into the ionisation chamber of an electron bombardment source through a nozzle,
   b) a fullerene ion beam is produced which is focused on a sample and scanned over an area of the sample
   c) a bend in the beam path acts to reject neutrals from the beam, and
   d) a means of mass filtering is used to select one fullerene from the mixture in the beam.
2) A fullerene ion gun as in claim (1) in which the mass filter is a Wien filter.
3) A fullerene ion gun as in claim (1) in which the mass filter is a dual pulser system.
4) A fullerene ion gun as in claims (1) to (3) in which the scanning is done by double deflection electrodes before the final lens.
5) A fullerene ion gun as in claims (1) to (4) in which the double deflection electrodes are successive and ratioed in length to form a compact assembly and to give a square, or near square, raster.
6) A fullerene ion gun as in claims (1) to (5) which includes an aperture selection mechanism.
7) A fullerene ion gun as in claims (1) to (6) which includes electrodes for alignment and for shaping of the beam.

INVESTOR IN PEOPLE

Application No:	GB 0126838.2	**Examiner:**	Geoff Holmes
Claims searched:	All	**Date of search:**	18 July 2003

Patents Act 1977 : Search Report under Section 17

Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
Y	All	US 4132892 A [WITTMAACK] see figure 1
A	-	EP 0501257 A1 [SHIMADZU] see figure 1
Y	All	Nuclear Instruments and Methods in Physics Research A Vol.415, No.3, October 1998 (Netherlands), S Majima et al., 'Production and acceleration of multiply charged C_{60} and C_{70} fullerene ions', pages 653-656.
Y	All	Molecular Materials Vol.11, No.1-2, 1998 (Switzerland), V Schäfer et al., 'Ionization and fragmentation of fullerene ions by electron impact', pages 131-134, especially page 131.
Y	All	Chinese Physics Letters Vol.10, No.8, 1993 (China), Fang Dufei et al., 'Production of C_{60}/C_{70} ion beams', pages 453-455, especially page 453.
Y	All	Zeitschrift für Physik D Vol.29, No.1, 1994 (Germany), D H Yu et al., 'Formation and fragmentation of C_n^+ clusters produced from a C_{60}/C_{70} mixture in an electron-impact ion source', pages 53-59, especially pages 53 & 54.
Y	All	Hyperfine Interactions Vol.99, No.1-3, February 1996 (Netherlands), S Matt et al., 'Production and decay of highly-charged fullerene ions', pages 175-191, esp. pages 176 & 177

Categories:

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

INVESTOR IN PEOPLE

Application No:	GB 0126838.2	**Examiner:**	Geoff Holmes
Claims searched:	All	**Date of search:**	18 July 2003

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC[V]:

H1D

Worldwide search of patent documents classified in the following areas of the IPC[7]:

H01J

The following online and other databases have been used in the preparation of this search report:

WPI, EPODOC, JAPIO

DERWENT-ACC-NO: 2003-715120

DERWENT-WEEK: 200368

TITLE: Fullerene ion gun for use in static time-of-flight secondary ion mass spectrometry, has nozzle for introducing fullerene vapor into ionization chamber to produce fullerene ion beam which is focussed on sample and scanned over area of sample

INVENTOR: BLENKINSOPP P W M; HILL R

PATENT-ASSIGNEE: IONOPTIKA LTD[IONON]

PRIORITY-DATA: 2001GB-026838 (November 8, 2001)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE
GB 2386747 A	September 24, 2003	EN

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
GB 2386747A	N/A	2001GB-026838	November 8, 2001

INT-CL-CURRENT:

TYPE	IPC DATE
CIPS	H01J49/14 20060101

ABSTRACTED-PUB-NO: GB 2386747 A

BASIC-ABSTRACT:

NOVELTY - In a fullerene ion gun, fullerene vapor is introduced directly into the ionization chamber of an electron bombardment source through a nozzle (5), a fullerene ion beam is produced which is focused on a sample and scanned over an area of the sample, a bend (11) in the path acts to reject neutrals from the beam, and a device for mass filtering is used to select one fullerene from the mixture in the beam.

USE - Used as a fullerene ion gun capable of producing a beam of C60 ions, which is pulsed, mass filtered and has sufficient intensity for use as a probe in static time-of-flight secondary ion mass spectrometry (S-TOF-SIMS).

ADVANTAGE - The source brightness is sufficient to allow the production of an ion beam with a sufficiently small spot size and sufficiently high current to be used in SIMS applications.

DESCRIPTION OF DRAWING(S) - The figure shows a side sectional view through a fullerene ion gun.

C60 powder (1)

Cylindrical reservoir (2)

Copper surround (3)

Heater (4)

Nozzle (5)

Grid (6)

Circular filament (7)

Extraction electrode (8)

Electrostatic lenses (9, 12)

Deflector plates (10)

1 degree bend (11)

Scanning electrodes (13)

Components for mass filtering (14)

Vacuum housing (15)

EQUIVALENT-ABSTRACTS:

INSTRUMENTATION AND TESTING

Preferred Ion Gun: The mass filter is a Wien filter, or a dual pulser system. The scanning is carried out by double deflection electrodes before the final lens. The double deflection electrodes are successive and ratioed in length to form a compact assembly and to give a square or near square raster. The ion gun comprises an aperture selection mechanism and electrodes for alignment and for shaping of the beam.

CHOSEN-DRAWING: Dwg.1/1

TITLE-TERMS: ION GUN STATIC TIME FLIGHT SECONDARY MASS SPECTROSCOPE NOZZLE INTRODUCING VAPOUR IONISE CHAMBER PRODUCE BEAM FOCUS SAMPLE SCAN AREA

DERWENT-CLASS: J04 S03 V05

CPI-CODES: J04-B01A;

EPI-CODES: S03-E10A3;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: 2003-196790

Non-CPI Secondary Accession Numbers: 2003-572019